Data Validation Checklist
Semivolatile Organic Analyses

Project:	35TH Avenue Superfund Site	Project No:	15268508.20000
Laboratory:	TestAmerica – Tampa, FL	Job ID.:	680-88766-2
Method:	SW-846 8270C Low-Level (PAH)	Associated Samples:	Refer to **Attachment A** (Sample Summary)
Matrix:	Soil and water	Date(s) Collected:	03/25/2013 and 03/26/2013
Reviewer:	Jane Lindsey	Date:	04/10/2013
Concurrence[1]:	Carol Lovett, Sarah Choyke	Date:	04/19/2013

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
1. Were sample storage and preservation requirements met? If temperature >6°C, then J/UJ-flag results.	✓				
2. Were all COC records signed and integrity seals intact, indicating that COC was maintained for all samples?	✓				
3. Were there any problems noted in laboratory data package concerning condition of samples upon receipt?		✓			
4. Do any soil samples contain more than 50% water? If yes, then results are to be reported on a wet-weight basis.		✓			
5. Were holding times met (≤7 and 14 days from collection to extraction for aqueous and solid samples, respectively; ≤40 days from extraction to analysis)? If not, then J/UJ-flag sample results. If grossly (2x) exceeded, then flag J/R.	✓				
6. Were results for all project-specified target analytes reported?	✓				
7. Were project-specified Reporting Limits achieved for undiluted sample analyses?	✓				
8. Were samples with analyte concentrations exceeding the calibration range of the instrument re-analyzed at a higher dilution? If not, then J-flag sample result.			✓		
9. Was a method blank extracted with each batch (i.e., one per 20 samples, per batch, per matrix and per level)?	✓				
10. Were target analytes detected in the method blank?		✓			
11. Were target analytes detected in equipment/rinsate blanks?		✓		PAH were not detected during the analysis of rinsate blank 032613-RB-Shovel (680-88766-23).	
12. Are equipment/rinsate blanks associated with every sample? If	✓			According to the QAPP, a rinsate blank is to be collected after each decontamination event, which	

[1] Independent technical reviewer

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
no, note in DV report.				occurs once per week per the client. A rinsate blank (032613-RB-Shovel) was collected during the week of 03/25/2013. The rinsate blank was analyzed for PAHs under this Test America Job ID.	
13. Were analytes detected in samples below the blank contamination action level? If yes, U-flag positive sample results <5x associated blank concentration (10x for common blank contaminants – phthalates)			✓	Blank contamination does not exist.	
14. Is a field duplicate associated with this Job?		✓			
15. Was precision deemed acceptable as defined by the project plans?			✓		
16. Were DFTPP ion abundance criteria (i.e., Table 3 of SW-846 8270C) met? If no, professional judgment may be applied to determine to what extent the data may be utilized.	✓			Alternate tuning criteria were used by the laboratory (i.e., EPA Method 525.2). All ion abundance criteria were met per EPA Method 525.2.	
17. Were samples analyzed within 12 hours of the DFTPP tune? If no, professional judgment may be applied to determine to what extent the data may be utilized.	✓				
18. Were initial and continuing calibration standards analyzed at the proper frequency for each instrument? • Ensure that a minimum of five standards are used for the initial calibration. If no, use professional judgment to determine the effect on the data and note in the reviewer narrative. • An initial calibration is to be associated with each sample analysis. • A continuing calibration standard is to be analyzed for every 12 hours of sample analysis per instrument.	✓			• Initial Calibration: 04/02/2013, instrument BSMC5973 • ICV: 04/02/2013 @ 15:34 • CCV: 04/03/2013 @ 11:45 • CCV: 04/05/2013 @ 12:15	
19. Were calibration results within laboratory/project specifications? • ICAL (Criteria: ≤15 mean %RSD with individual CCC %RSD ≤30 (≤50% for poor performers), OR r≥0.995, OR r^2≥0.99, and RRF ≥0.050 (≥0.010 for poor performers)): o If %RSD>15 (>50% for poor performers), or r <0.995, or r^2 <0.995, then J-flag positive results and UJ-flag non-detects o If mean RRF <0.050 (<0.010 for poor performers), then J-flag positive results and R-flag non-detects • ICV and CCV (Criteria: ≤20%D (≤50% for poor performers) and RF ≥0.050 (≥0.010 for poor performers)):		✓		ICV of 04/02/2013 @ 15:34, instrument BSMC5973: • Pyrene @ -21.4%D (Lab: ≤35, Project: ≤20), 78.5%R • Chrysene @ -23.5%D (Lab: ≤35, Project: ≤20), 76.5%R • Benzo(b)fluoranthene @ -21.1%D (Lab: ≤35, Project: ≤20), 79%R • Benzo(a)pyrene @ -24.3%D (Lab: ≤35, Project: ≤20), 75.5%R A negative bias is indicated by the ICV percent difference, therefore, J and UJ flag results in associated	J, UJ

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
○ If %D>20 (>50% for poor performers), then J-flag positive results and UJ-flag non-detects ○ If RF <0.050 (<0.010 for poor performers), then UJ-flag non-detected semivolatile target compounds				samples[2].	
20. Was a LCS prepared for each batch and matrix?	✓				
21. Were LCS recoveries within lab control limits? If no, J-flag positive results when %R >Upper Control Limit (UCL) and J/R-flag results when %R <Lower Control Limit (LCL).	✓				
22. Were LCS/LCSD RPD within lab specifications? If no, J-flag positive results and UJ-flag non-detects	✓			LCSD associated with water prep batch 136013.	
23. Was a MS/MSD pair extracted at the proper frequency (one per 20 samples per batch)?	✓			• Water Prep Batch 136013: 680-88766-23 (032613-RB-Shovel), MS only due to limited sample volume. A LCSD analysis was conducted in lieu of the MSD. • Soil Prep Batch 136063: 680-88766-21 (CV0014AB-GS), MS/MSD	
24. Is the MS/MSD parent sample a project-specific sample?	✓			See above.	
25. Were MS/MSD recoveries within laboratory/project specifications? *Only QC results for project samples that are reported under this Job ID are evaluated.* • If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. • If either MS or MSD recovery meets control limits, qualification of data is not warranted. • MS and MSD %R<10: J and R Flag positive and ND results, respectively • MS and MSD %R >10 and <LCL: J-Flag positive and UJ-flag non-detect results • MS and MSD R% >UCL (or 140): J-Flag positive results		✓		032613-RB-Shovel (680-88766-23): Refer to **Attachment B**, MS recoveries. UJ Flag results due to low MS recoveries.	UJ
26. Were laboratory criteria met for precision during the MS/MSD analysis? *Only QC results for project samples that are reported under this Job ID are evaluated.* • If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. • If %RPD > UCL, J-flag positive result and UJ-flag non-detect result.	✓				

[2] 680-88766-21 and 23

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
27. Were surrogate recoveries within lab/project specifications? • If %R <10, then J-flag positive and R-flag non-detect associated sample results • If %R >UCL, then J-flag positive results • %R ≥10%, but <LCL, then J-flag positive results and UJ-flag non-detect results • If 1 %R >UCL and 1 %R ≥10%, but <LCL, then J-flag positive results and UJ-flag non-detect results	✓				
28. Were internal standard (IS) results within lab/project specifications? • If IS area counts are less than 50% of the midpoint calibration standard, then J-flag positive and UJ-flag non-detect associated sample results • If IS area counts are greater than 100% of the midpoint calibration standard, then J-flag positive results • If extremely low area counts are reported or performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated, J-flag positive and R-flag non-detect results • If retention time of sample's internal standard is not within 30 seconds of the associated calibration standard, R-flag associated data. • The chromatographic profile for that sample must be examined to determine if any false positives or negatives exists. For shifts of large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results need not be qualified as R, if mass spectral criteria are met.	✓				
29. Were lab comments included in report?	✓			Refer to **Attachment C** (Case Narrative)	

Comments: The data validation was conducted in accordance with the *Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1* (OTIE, October 2012). The data review process was modeled after the *USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG) for Organic Methods Data Review* (EPA, October 1999) and *USEPA CLP NFG for Low Concentration Organic Methods Data Review* (EPA, June 2001). Sample results have been qualified based on the results of the data review process (**Attachment D**). Criteria for acceptability of data were based upon available site information, analytical method requirements, guidance documents, and professional judgment.

Data Validation Checklist (Continued)

DV Flag Definitions:

J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
R	The sample results are unusable. The analyte may or may not be present in the sample.
U	The analyte was analyzed for, but was not detected above the associated level; blank contamination may exist.
UJ	The analyte was not detected above the limit, and the limit is approximate and may be inaccurate or imprecise.

ATTACHMENT A

SAMPLE SUMMARY

Sample Summary

Client: Oneida Total Integrated Enterprises LLC
Project/Site: 35th Avenue Superfund Site

TestAmerica Job ID: 680-88766-2
SDG: 68088766-2

Lab Sample ID	Client Sample ID	Matrix	Collected	Received
680-88766-21	CV0014AB-GS	Solid	03/25/13 15:18	03/28/13 09:37
680-88766-23	032613-RB-shovel	Water	03/26/13 13:00	03/28/13 09:37

ATTACHMENT B

MS RESULTS

FORM III
GC/MS SEMI VOA MATRIX SPIKE RECOVERY

Lab Name: TestAmerica Tampa Job No.: 680-88766-2

SDG No.: 68088766-2

Matrix: Water Level: Low Lab File ID: 1CD05007.D

Lab ID: 680-88766-23 MS Client ID: 032613-RB-shovel MS

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC	QC LIMITS REC	#	Action
Acenaphthene	10.4	2.5 U	4.74	45	55-132	F	UJ ND
Acenaphthylene	10.4	1.3 U	5.18	50	39-130		
Anthracene	10.4	0.25 U	4.97	48	39-130		
Benzo[a]anthracene	10.4	0.25 U	4.72	45	54-135	F	UJ ND
Benzo[a]pyrene	10.4	0.25 U	2.13	20	21-130	F	
Benzo[b]fluoranthene	10.4	0.25 U	2.68	26	37-130	F	
Benzo[g,h,i]perylene	10.4	0.63 U	1.33	13	26-130	F	
Benzo[k]fluoranthene	10.4	0.25 U	2.38	23	38-130	F	
Chrysene	10.4	0.25 U	3.55	34	56-130	F	
Dibenz(a,h)anthracene	10.4	0.25 U	1.12	11	13-130	F	
Fluoranthene	10.4	0.63 U	5.46	52	60-130	F	
Fluorene	10.4	2.5 U	4.83	46	55-140	F	
Indeno[1,2,3-cd]pyrene	10.4	0.25 U	1.28	12	21-130	F	
1-Methylnaphthalene	10.4	2.5 U	5.64	54	49-130		
2-Methylnaphthalene	10.4	2.5 U	4.68	45	48-130	F	UJ ND
Naphthalene	10.4	2.5 U	5.39	52	54-133	F	
Phenanthrene	10.4	0.63 U	5.42	52	60-136	F	
Pyrene	10.4	0.63 U	5.24	50	60-138	F	

Column to be used to flag recovery and RPD values

FORM III 8270C LL

ATTACHMENT C

CASE NARRATIVE

Job ID: 680-88766-2

Laboratory: TestAmerica Savannah

Narrative

CASE NARRATIVE

Client: Oneida Total Integrated Enterprises LLC

Project: 35th Avenue Superfund Site

Report Number: 680-88766-2

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

RECEIPT

The samples were received on 03/28/2013; the samples arrived in good condition, properly preserved and on ice. The temperature of the coolers at receipt was 1.4 C.

SEMIVOLATILE ORGANIC COMPOUNDS BY GCMS - LOW LEVEL

Sample CV0014AB-GS (680-88766-21) was analyzed for Semivolatile Organic Compounds by GCMS - Low Level in accordance with EPA SW-846 Method 8270C. The samples were prepared on 04/02/2013 and analyzed on 04/03/2013.

Sample CV0014AB-GS (680-88766-21)[4X] required dilution prior to analysis. The reporting limits have been adjusted accordingly.

No difficulties were encountered during the SVOAs analysis.

All quality control parameters were within the acceptance limits.

SEMIVOLATILE ORGANIC COMPOUNDS (GC-MS)-WATER

Sample 032613-RB-shovel (680-88766-23) was analyzed for semivolatile organic compounds (GC-MS) in accordance with EPA SW-846 Method 8270C. The samples were prepared on 04/02/2013 and analyzed on 04/05/2013.

Several analytes recovered outside the recovery criteria low for the MS of sample 032613-RB-shovel (680-88766-23) in batch 660-136171.

No other difficulties were encountered during the semivolatiles analysis.

All other quality control parameters were within the acceptance limits.

ATTACHMENT D

QUALIFIED SAMPLE RESULTS

Client Sample ID: CV0014AB-GS

Lab Sample ID: 680-88766-21

Date Collected: 03/25/13 15:18
Matrix: Solid
Date Received: 03/28/13 09:37
Percent Solids: 73.9

Method: 8270C LL - Semivolatile Organic Compounds by GCMS - Low Levels

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Acenaphthene	530	U	530	110	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Acenaphthylene	**34**	**J**	210	27	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Anthracene	45	U J *	45	22	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Benzo[a]anthracene	**200**		43	21	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Benzo[a]pyrene	**100**	J	55	28	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Benzo[b]fluoranthene	**150**	J	65	32	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Benzo[g,h,i]perylene	**130**		110	23	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Benzo[k]fluoranthene	**79**		43	19	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Chrysene	**210**	J	48	24	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Dibenz(a,h)anthracene	**62**	**J**	110	22	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Fluoranthene	**210**		110	21	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Fluorene	**39**	**J**	110	22	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Indeno[1,2,3-cd]pyrene	**76**	**J**	110	38	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
1-Methylnaphthalene	**120**	**J**	210	23	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
2-Methylnaphthalene	**130**	**J**	210	38	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Naphthalene	**100**	**J**	210	23	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Phenanthrene	**200**		43	21	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4
Pyrene	**210**	J	110	20	ug/Kg	☼	04/02/13 11:33	04/03/13 16:10	4

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
o-Terphenyl	*95*		*30 - 130*	*04/02/13 11:33*	*04/03/13 16:10*	*4*

Client Sample ID: 032613-RB-shovel

Lab Sample ID: 680-88766-23

Date Collected: 03/26/13 13:00
Matrix: Water
Date Received: 03/28/13 09:37

Method: 8270C LL - Semivolatile Organic Compounds by GCMS - Low Levels

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Acenaphthene	2.5	U F J	2.5	0.63	ug/L		04/02/13 08:08	04/05/13 12:54	1
Acenaphthylene	1.3	U	1.3	0.31	ug/L		04/02/13 08:08	04/05/13 12:54	1
Anthracene	0.25	U J *	0.25	0.095	ug/L		04/02/13 08:08	04/05/13 12:54	1
Benzo[a]anthracene	0.25	U F J	0.25	0.063	ug/L		04/02/13 08:08	04/05/13 12:54	1
Benzo[a]pyrene	0.25	U F J	0.25	0.071	ug/L		04/02/13 08:08	04/05/13 12:54	1
Benzo[b]fluoranthene	0.25	U F J	0.25	0.063	ug/L		04/02/13 08:08	04/05/13 12:54	1
Benzo[g,h,i]perylene	0.63	U F J	0.63	0.13	ug/L		04/02/13 08:08	04/05/13 12:54	1
Benzo[k]fluoranthene	0.25	U F J	0.25	0.071	ug/L		04/02/13 08:08	04/05/13 12:54	1
Chrysene	0.25	U F J	0.25	0.086	ug/L		04/02/13 08:08	04/05/13 12:54	1
Dibenz(a,h)anthracene	0.25	U F J	0.25	0.063	ug/L		04/02/13 08:08	04/05/13 12:54	1
Fluoranthene	0.63	U F J	0.63	0.068	ug/L		04/02/13 08:08	04/05/13 12:54	1
Fluorene	2.5	U F J	2.5	0.63	ug/L		04/02/13 08:08	04/05/13 12:54	1
Indeno[1,2,3-cd]pyrene	0.25	U F J	0.25	0.063	ug/L		04/02/13 08:08	04/05/13 12:54	1
1-Methylnaphthalene	2.5	U	2.5	0.63	ug/L		04/02/13 08:08	04/05/13 12:54	1
2-Methylnaphthalene	2.5	U F J	2.5	0.63	ug/L		04/02/13 08:08	04/05/13 12:54	1
Naphthalene	2.5	U F J	2.5	0.31	ug/L		04/02/13 08:08	04/05/13 12:54	1
Phenanthrene	0.63	U F J	0.63	0.25	ug/L		04/02/13 08:08	04/05/13 12:54	1
Pyrene	0.63	U F J	0.63	0.11	ug/L		04/02/13 08:08	04/05/13 12:54	1

Surrogate	*%Recovery*	*Qualifier*	*Limits*	*Prepared*	*Analyzed*	*Dil Fac*
o-Terphenyl	*67*		*30 - 130*	*04/02/13 08:08*	*04/05/13 12:54*	*1*

* Flagging error, M Meyers-Lee, 4/19/2013